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IS 6092-3-5 (2004): Methods of Sampling and Test for Fertilizers, Part 3: Determination of Phosphorus, Section 5: Extraction of Phosphates Soluble in Mineral Acids [FAD 7: Soil Quality and Gertilizers]



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भारतीय मानक
उर्वरकों के नमूने लेना और परीक्षण पद्धतियाँ

भाग 3 फास्फोरस ज्ञात करना

अनुभाग 5 खनिज अम्ल में फास्फोरस विलेय का निष्कर्षण

Indian Standard
**METHODS OF SAMPLING AND TEST FOR
FERTILIZERS**

PART 3 DETERMINATION OF PHOSPHORUS

Section 5 Extraction of Phosphates Soluble in Mineral Acids

ICS 65.080

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 1

NATIONAL FOREWORD

This Indian Standard (Part 3/Sec 5) which is identical with ISO 7497 : 1984 'Fertilizers — Extraction of phosphates soluble in mineral acids' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Fertilizers Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 3 DETERMINATION OF PHOSPHORUS

Section 5 Extraction of Phosphates Soluble in Mineral Acids

0 Introduction

The two methods specified in this International Standard have been recognized as equivalent for the extraction of mineral acid-soluble phosphates.

1 Scope and field of application

This International Standard specifies a method for the extraction of mineral acid-soluble phosphates by attack with a mixture of hydrochloric and nitric acids and a method by attack with a mixture of sulfuric and nitric acids.

These methods are applicable to all phosphate fertilizers and to mineral phosphates containing low amounts of organic matter.

2 Method A: Hydrochloric acid/nitric acid attack

2.1 Principle

Dissolution of the phosphates present in fertilizers or in mineral phosphates in a mixture of hydrochloric and nitric acids.

2.2 Reagents

All reagents shall be of recognized analytical grade, and the water used shall be distilled water or demineralized water of equivalent purity.

Acid mixture.

Add 3 volumes of nitric acid ($\rho_{20} = 1,40$ g/ml) to 1 volume of hydrochloric acid ($\rho_{20} = 1,19$ g/ml). Mix well, dilute with four volumes of water and mix again.

Use a freshly prepared acid mixture.

2.3 Apparatus

Ordinary laboratory apparatus, and in particular:

2.3.1 Grinder.

2.3.2 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of ISO 1042, class A.

2.4 Test sample

Grind the laboratory sample until it passes through a sieve of aperture size 0,5 mm.

2.5 Procedure

2.5.1 Test portion

Weigh, to the nearest 1 mg, 2,5 g of the test sample and transfer it to a 400 ml beaker.

2.5.2 Extraction

Add 50 ml of the acid mixture (2.2). Cover the beaker with a clock-glass. Bring the mixture to boiling and simmer for 30 min. Add 100 ml of water, bring to boiling and simmer for a further 15 min. Cool to room temperature.

Transfer to the one-mark volumetric flask (2.3.2) and dilute to the mark with water. Mix carefully and filter the solution through a dry, folded medium-grade filter paper, free from phosphates. Discard the first two approximately 30 ml portions.

NOTE — The filtrate should be clear.

3 Method B: Sulfuric acid/nitric acid attack

3.1 Principle

Dissolution of the phosphates present in fertilizers or in mineral phosphates in a mixture of sulfuric and nitric acids.

3.2 Reagents

All reagents shall be of recognized analytical grade, and the water used shall be distilled water or demineralized water of equivalent purity.

3.2.1 Sulfuric acid ($\rho_{20} = 1,84$ g/ml).

3.2.2 Nitric acid ($\rho_{20} = 1,40$ g/ml).

3.3 Apparatus

Ordinary laboratory apparatus, and in particular:

3.3.1 Kjeldahl flask, of capacity at least 500 ml, or a flask, of capacity 250 ml, fitted with a reflux condenser.

3.3.2 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of ISO 1042, class A.

3.4 Procedure

3.4.1 Test portion

Weigh, to the nearest 1 mg, 2,5 g of the test sample and transfer it to the Kjeldahl flask (3.3.1).

3.4.2 Extraction

Add 15 ml of water and mix so as to bring the test portion into suspension. Add 20 ml of nitric acid (3.2.2) and, carefully, 30 ml of the sulfuric acid (3.2.1).

As soon as initial violent reaction has ceased, bring the contents of the flask slowly to the boil and boil for 30 min. Allow to cool and then add, carefully and with mixing, about 100 ml of water. Bring to the boil again and boil for 15 min.

Cool and transfer the liquid quantitatively to the one-mark volumetric flask (3.3.2). Make up to the mark with water, mix and filter through a dry, folded filter paper, free from phosphates, rejecting the first portion of filtrate.

4 Test report

The test report for the subsequent determination of the phosphates content shall include the following information relevant to the preparation of the test solution:

- a) all the information necessary for the complete identification of the sample;
- b) a reference to this International Standard and the method used;
- c) any operations not specified in this International Standard, or regarded as optional, as well as any incident likely to affect the results of the determination of water-soluble phosphates content.

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